

# STIC Search Report

## Biotech-Chem Library

STIC Database Tracking Number: 188062

**TO:** Rei-Tsang Shiao  
**Location:** REM-5A10/5C18  
**Art Unit:** 1626  
**Friday, May 12, 2006**

**Case Serial Number:** 10/811727

**From:** Paul Schulwitz  
**Location:** Biotech-Chem Library  
**REM-1A65**  
**Phone:** 571-272-2527

**Paul.schulwitz@uspto.gov**

### Search Notes

Examiner Shiao,

Please review the attached search results.

If you have any questions or if you would like to refine the search query, please feel free to contact me at any time.

Thank you for using STIC search services!

Paul Schulwitz  
Technical Information Specialist  
REM-1A65  
571-272-2527



# STIC SEARCH RESULTS FEEDBACK FORM

## Biotech-Chem Library

Questions about the scope or the results of the search? Contact **the searcher or contact:**

**Mary Hale, Information Branch Supervisor**  
Remsen Bldg. 01 D86  
571-272-2507

## Voluntary Results Feedback Form

- *I am an examiner in Workgroup:*  *Example: 1610*
- *Relevant prior art found, search results used as follows:*
- 102 rejection
  - 103 rejection
  - Cited as being of interest.
  - Helped examiner better understand the invention.
  - Helped examiner better understand the state of the art in their technology.

*Types of relevant prior art found:*

- Foreign Patent(s)
- Non-Patent Literature  
(journal articles, conference proceedings, new product announcements etc.)

➤ *Relevant prior art not found:*

- Results verified the lack of relevant prior art (helped determine patentability).
- Results were not useful in determining patentability or understanding the invention.

**Comments:**

Drop off or send completed forms to STIC-Biotech-Chem Library Remsen Bldg.



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188062 ACCESS DB # \_\_\_\_\_  
PLEASE PRINT CLEARLY

Scientific and Technical Information Center

SEARCH REQUEST FORM

Requester's Full Name: Robert (Reiley) Shioi Examiner #: 79521 Date: 5/1/06  
Art Unit: 1626 Phone Number: 20707 Serial Number: 1081727  
Location (Bldg/Room#): RPPM (Mailbox #) 5A10X Results Format Preferred (circle): PAPER DISK  
\*\*\*\*\*  
/5C18

To ensure an efficient and quality search, please attach a copy of the cover sheet, claims, and abstract or fill out the following:

Title of Invention: Gamma crystalline form  
Inventors (please provide full names): Pfleiffer et al.

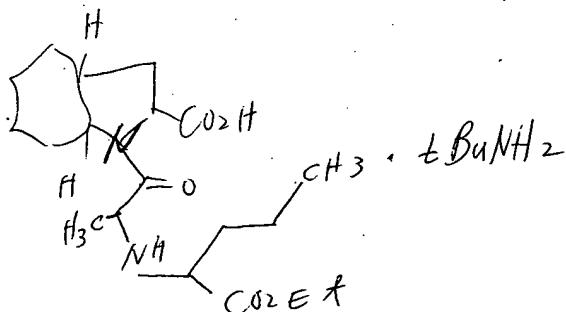
Earliest Priority Date: \_\_\_\_\_

Search Topic:

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc., if known.

\*For Sequence Searches Only\* Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

I. search cpd 2 (see claim 12)



II. search crystalline form of cpd 2.

INV  
L15  
L30  
WR

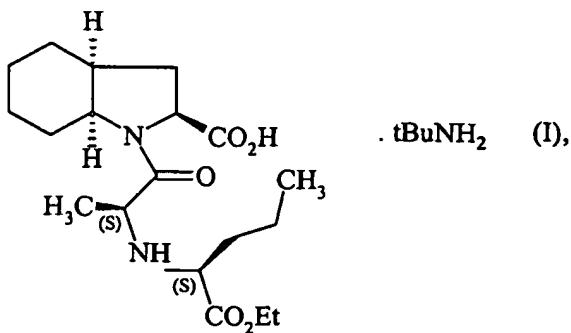
30 531.5  
30

STAFF USE ONLY		Type of Search	Vendors and cost where applicable		
Searcher:	_____	NA Sequence (#)	_____	STN	_____ Dialog
Searcher Phone #:	_____	AA Sequence (#)	_____	Questel/Orbit	_____ Lexis/Nexis
Searcher Location:	_____	Structure (#)	_____	Westlaw	_____ WWW/Internet
Date Searcher Picked Up:	_____	Bibliographic	In-house sequence systems		
Date Completed:	_____	Litigation	_____ Commercial	_____ Oligomer	_____ Score/Length
Searcher Prep & Review Time:	_____	Fulltext	_____ Interference	_____ SPDI	_____ Encode/Transl
Online Time:	_____	Other	Other (specify)		

LISTING OF CLAIMS

1-11. (canceled)

12. (previously presented) A  $\gamma$  crystalline form of the compound of formula (I) :



exhibiting essentially the following powder X-ray diffraction data, measured using a diffractometer (copper anticathode) and expressed in terms of inter-planar distance  $d$ , Bragg's angle 2 theta, intensity and relative intensity (expressed as a percentage with respect to the most intense ray) :

10

Angle 2 theta ( $^{\circ}$ )	Inter-planar distance $d$ ( $\text{\AA}$ )	Intensity	Relative intensity (%)
6.298	14.02	630	39.8
7.480	11.81	380	24
8.700	10.16	1584	100
9.276	9.53	318	20.1
10.564	8.37	526	33.2
11.801	7.49	54	3.4
12.699	6.96	86	5.4
13.661	6.48	178	11.2
14.095	6.28	163	10.3
14.332	6.17	290	18.3
14.961	5.92	161	10.2
15.793	5.61	128	8.1
16.212	5.46	179	11.3
16.945	5.23	80	5.1

17.291	5.12	92	5.8
17.825	4.97	420	26.5
18.100	4.90	159	10
18.715	4.74	89	5.6
19.017	4.66	118	7.4
19.362	4.58	134	8.5
19.837	4.47	133	8.4
20.609	4.31	95	6
21.232	4.18	257	16.2
21.499	4.13	229	14.5
21.840	4.07	127	8
22.129	4.01	191	12.1
22.639	3.92	137	8.6
23.000	3.86	88	5.6
23.798	3.74	147	9.3
24.170	3.68	70	4.4
25.066	3.55	167	10.5
25.394	3.50	165	10.4
26.034	3.42	84	5.3
26.586	3.35	75	4.7
27.541	3.24	74	4.7
28.330	3.15	85	5.4
29.589	3.02	96	6.1

13. (previously presented) A process for the preparation of the  $\gamma$  crystalline form of the compound of claim 12, wherein a solution of perindopril tert-butylamine salt in chloroform is heated at reflux, the solution is then cooled to 0°C and the solid obtained is collected by filtration.
- 5 14. (previously presented) A process for the preparation of the  $\gamma$  crystalline form of the compound of claim 12, wherein a solution of perindopril tert-butylamine salt in ethyl acetate is heated at reflux, the solution is rapidly cooled, the solid thereby obtained is then collected by filtration, it is suspended in chloroform, the suspension is stirred at ambient temperature for 5 to 10 days, and the solid is then collected by filtration.
- 10 15. (previously presented) The process of claim 13, wherein the compound of formula (I) obtained by the preparation process described in patent specification EP 0 308 341 is used.

16. (previously presented) The process of claim 13, wherein the concentration of the compound of formula (I) in the chloroform is 150 to 300 g/litre.
17. (previously presented) The process of claim 14, wherein the compound of formula (I) obtained by the preparation process described in patent specification EP 0 308 341 is used.
18. (previously presented) The process according to claim 14, wherein the concentration of the compound of formula (I) in the ethyl acetate is 70 to 90 g/litre.

5

19. (previously presented) A method of treating a living animal body afflicted with a condition requiring an inhibitor of angiotensin I converting enzyme, comprising the step of administering to the living animal body an amount of the compound of claim 12 which is effective for alleviation of the condition.
- 10 20. (previously presented) A pharmaceutical composition comprising, as active principle, an effective amount of the compound of claim 12, together with one or more pharmaceutically acceptable excipients or vehicles.
- 15 21. (previously presented) A method of treating a living animal body afflicted with a cardiovascular disease, comprising the step of administering to the living animal body an amount of the compound of claim 12 which is effective for alleviation of the condition.
22. (previously presented) The pharmaceutical composition of claim 20, which also comprises a diuretic.
- 20 23. (previously presented) The pharmaceutical composition of claim 22, wherein the diuretic is indapamide.



## UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE  
 United States Patent and Trademark Office  
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 P.O. Box 1450  
 Alexandria, Virginia 22313-1450  
[www.uspto.gov](http://www.uspto.gov)



Bib Data Sheet

CONFIRMATION NO. 1430

SERIAL NUMBER 10/811,727	FILING DATE 03/29/2004 RULE	CLASS 548	GROUP ART UNIT 1626	ATTORNEY DOCKET NO. SERVIER 398 PCT
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## APPLICANTS

Bruno Pfeiffer, Saint Leu La Foret, FRANCE;

Yves-Michel Ginot, Orleans, FRANCE;  
Gerard Coquerel, Boos, FRANCE; Stephane Beilles, Dijon, FRANCE;

## \*\* CONTINUING DATA \*\*\*\*\*

This application is a CON of 10/312,903 12/31/2002 ABN

## \*\* FOREIGN APPLICATIONS \*\*\*\*\*

FRANCE 00.08791 07/06/2000

FRANCE PCT/FR01/02169 07/06/2001

## IF REQUIRED, FOREIGN FILING LICENSE GRANTED

\*\* 06/07/2004

Foreign Priority claimed 35 USC 119 (a-d) conditions met	yes <input checked="" type="checkbox"/> no	STATE OR COUNTRY	SHEETS DRAWING 0	TOTAL CLAIMS 12	INDEPENDENT CLAIMS 1
Verified and Acknowledged Examiner's Signature	yes <input checked="" type="checkbox"/> no <input type="checkbox"/> MCA after Allowance Initials	FRANCE			

## ADDRESS

The Firm of Hueschen and Sage  
 500 Columbia Plaza  
 350 East Michigan Avenue  
 Kalamazoo, MI  
 49007

## TITLE

Gamma crystalline form of perindopril tert-butylamine salt

FILING FEE	FEES: Authority has been given in Paper	<input type="checkbox"/> All Fees <input type="checkbox"/> 1.16 Fees ( Filing ) <input type="checkbox"/> 1.17 Fees ( Processing Ext. of )
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# Application Search

Shiao 10/811,727

05/12/2006

L3 ANSWER 1 OF 1 HCAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 2001:816626 HCAPLUS  
 DOCUMENT NUMBER: 135:344373  
 ENTRY DATE: Entered STN: 09 Nov 2001  
 TITLE: Process for preparing the novel  $\gamma$  crystalline  
 form of the diuretic perindopril tert-butylamine salt  
 INVENTOR(S): Pfeiffer, Bruno; Ginot, Yves-Michel; Coquerel, Gerard;  
 Beilles, Stephane  
 PATENT ASSIGNEE(S): Adir et Compagnie, Fr.  
 SOURCE: PCT Int. Appl., 11 pp.  
 CODEN: PIXXD2  
 DOCUMENT TYPE: Patent  
 LANGUAGE: French  
 INT. PATENT CLASSIF.:  
     MAIN: C07D  
 CLASSIFICATION: 27-11 (Heterocyclic Compounds (One Hetero Atom))  
 Section cross-reference(s): 63, 75  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001083439	A2	20011108	WO 2001-FR2169	20010706
WO 2001083439	A3	20020207		
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
FR 2811318	A1	20020111	FR 2000-8791	20000706
FR 2811318	B1	20020823		
CA 2415447	AA	20011108	CA 2001-2415447	20010706
AU 2001076420	A5	20011112	AU 2001-76420	20010706
EP 1296948	A2	20030402	EP 2001-954060	20010706
EP 1296948	B1	20030910		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
BR 2001012211	A	20030506	BR 2001-12211	20010706
AT 249435	E	20030915	AT 2001-954060	20010706
JP 2003531890	T2	20031028	JP 2001-580868	20010706
JP 3592296	B2	20041124		
PT 1296948	T	20031231	PT 2001-954060	20010706
ES 2206423	T3	20040516	ES 2001-1954060	20010706
NZ 523311	A	20040625	NZ 2001-523311	20010706
EE 200300003	A	20040816	EE 2003-3	20010706
US 2003158121	A1	20030821	US 2002-312903	20021231
ZA 2003000025	A	20040210	ZA 2003-25	20030102
NO 2003000051	A	20030106	NO 2003-51	20030106
BG 107534	A	20031231	BG 2003-107534	20030205
HR 2003000078	A1	20030430	HR 2003-78	20030206
HR 20030078	B1	20040630		
US 2004248817	A1	20041209	US 2004-811727	20040329 <--
JP 2005002120	A2	20050106	JP 2004-206157	20040713
PRIORITY APPLN. INFO.:			FR 2000-8791	A 20000706

JP 2001-580868	A3 20010706
WO 2001-FR2169	W 20010706
US 2002-312903	B1 20021231

## PATENT CLASSIFICATION CODES:

PATENT NO.	CLASS	PATENT FAMILY CLASSIFICATION CODES
WO 2001083439	ICM	C07D
	IPCI	C07D [ICM, 7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
	ECLA	C07D209/42
FR 2811318	IPCI	C07D0209-42 [ICM, 7]; A61K0031-405 [ICS, 7]; A61P0009-00 [ICS, 7]; A61P0007-10 [ICS, 7]; A61K0031-405 [ICI, 7]; A61K0031-4045 [ICI, 7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
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BR 2001012211	IPCI	C07D0209-42 [ICM, 7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
AT 249435	IPCI	C07D0209-42 [ICM, 7]
JP 2003531890	IPCI	C07D0209-42 [ICM, 7]; A61K0031-404 [ICS, 7]; A61K0045-00 [ICS, 7]; A61P0009-10 [ICS, 7]; A61P0013-02 [ICS, 7]; A61P0043-00 [ICS, 7]; B01D0009-02 [ICS, 7]; C07M0007-00 [ICS, 7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
PT 1296948	IPCI	C07D0209-42 [ICM, 7]
ES 2206423	IPCI	C07D0209-42 [ICM, 7]
NZ 523311	IPCI	C07D0209-42 [ICM, 7]; A61K0031-404 [ICS, 7]; A61P0009-00 [ICS, 7]
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EE 200300003	IPCI	A61K0031-404 [ICM, 7]; A61P0009-00 [ICS, 7]; C07D0209-42 [ICS, 7]
US 2003158121	IPCI	A61K0038-04 [ICM, 7]; C07D0209-12 [ICS, 7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
	NCL	514/019.000
	ECLA	C07D209/42
ZA 2003000025	IPCI	C07D [ICM, 7]; A61K [ICS, 7]; A61P [ICS, 7]
NO 2003000051	IPCI	C07D0207-16 [ICM, 7]
BG 107534	IPCI	C07D0209-42 [ICM, 7]
HR 2003000078	IPCI	C07D0209-42 [ICM, 7]; A61K0031-404 [ICS, 7]; A61P0009-00 [ICS, 7]
US 2004248817	IPCI	A61K0038-04 [ICM, 7]; C07K0005-04 [ICS, 7]; C07D0209-42 [ICS, 7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
	NCL	514/019.000
	ECLA	C07D209/42
JP 2005002120	IPCI	C07K0005-062 [ICM, 7]; A61K0031-404 [ICS, 7]; A61K0038-00 [ICS, 7]; A61K0045-00 [ICS, 7]; A61P0007-10 [ICS, 7]; A61P0009-00 [ICS, 7]; A61P0009-04 [ICS, 7]; A61P0009-12 [ICS, 7]; A61P0043-00 [ICS, 7]; C07D0209-08 [ICS, 7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
	FTERM	4C084/AA02; 4C084/AA03; 4C084/AA07; 4C084/AA19; 4C084/BA01; 4C084/BA14; 4C084/BA23; 4C084/CA59; 4C084/MA02; 4C084/NA14; 4C084/ZA361; 4C084/ZA362; 4C084/ZA421; 4C084/ZA422; 4C084/ZA832; 4C084/ZC202;

4C086/AA01; 4C086/AA02; 4C086/BC13; 4C086/MA02;  
 4C086/MA04; 4C086/NA05; 4C086/ZA36; 4C086/ZA42;  
 4C086/ZA83; 4C204/CB03; 4C204/DB03; 4C204/EB02;  
 4C204/FB35; 4C204/GB01; 4H045/AA10; 4H045/AA30;  
 4H045/BA11; 4H045/DA57; 4H045/EA23

## ABSTRACT:

The  $\gamma$  crystalline form of the diuretic perindopril tert-butylamine salt (I) is prepared by refluxing a chloroform-I solution, cooling the solution to 0°, and filtering the I  $\gamma$  crystal modification which is characterized by its X-ray diffraction pattern; a I-containing formulation is presented.

SUPPL. TERM: diuretic perindopril tertiary butylamine salt crystal modification

INDEX TERM: Polymorphism (crystal)  
 (process for preparing the novel  $\gamma$  crystalline form of the diuretic perindopril tert-butylamine salt)

INDEX TERM: Cardiovascular agents  
 (process for preparing the novel  $\gamma$  crystalline form of the diuretic perindopril tert-butylamine salt for preparation of)

INDEX TERM: Cooling  
 Crystallization  
 Filtration  
 (process for preparing the novel  $\gamma$  crystalline form of the diuretic perindopril tert-butylamine salt using)

INDEX TERM: Separation  
 (reflux; process for preparing the novel  $\gamma$  crystalline form of the diuretic perindopril tert-butylamine salt using)

INDEX TERM: 9015-82-1  
 ROLE: BSU (Biological study, unclassified); BUU (Biological use, unclassified); BIOL (Biological study); USES (Uses)  
 (inhibitors; process for preparing the novel  $\gamma$  crystalline form of the diuretic perindopril tert-butylamine salt for preparation of)

INDEX TERM: 107133-36-8  
 ROLE: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)  
 (process for preparing the novel  $\gamma$  crystalline form of the diuretic perindopril tert-butylamine salt)

INDEX TERM: 484-42-4  
 ROLE: BPR (Biological process); BSU (Biological study, unclassified); BIOL (Biological study); PROC (Process)  
 (process for preparing the novel  $\gamma$  crystalline form of the diuretic perindopril tert-butylamine salt as an intermediate in the preparation of inhibitors of)

INDEX TERM: 26807-65-8P, Indapamide  
 ROLE: PNU (Preparation, unclassified); PREP (Preparation)  
 (process for preparing the novel  $\gamma$  crystalline form of the diuretic perindopril tert-butylamine salt for the preparation of)

INDEX TERM: 67-66-3, Chloroform, uses 141-78-6, Ethyl acetate, uses  
 ROLE: NUU (Other use, unclassified); USES (Uses)  
 (solvent; process for preparing the novel  $\gamma$  crystalline form of the diuretic perindopril tert-butylamine salt)

IT 9015-82-1  
 RL: BSU (Biological study, unclassified); BUU (Biological use, unclassified); BIOL (Biological study); USES (Uses)  
 (inhibitors; process for preparing the novel  $\gamma$  crystalline form of the diuretic perindopril tert-butylamine salt for preparation of)

RN 9015-82-1 HCPLUS  
 CN Carboxypeptidase, dipeptidyl, A (9CI) (CA INDEX NAME)

\*\*\* STRUCTURE DIAGRAM IS NOT AVAILABLE \*\*\*

IT 107133-36-8

RL: PEP (Physical, engineering or chemical process); PRP (Properties);  
 PROC (Process)  
 (process for preparing the novel  $\gamma$  crystalline form of the diuretic  
 perindopril tert-butylamine salt)

RN 107133-36-8 HCPLUS

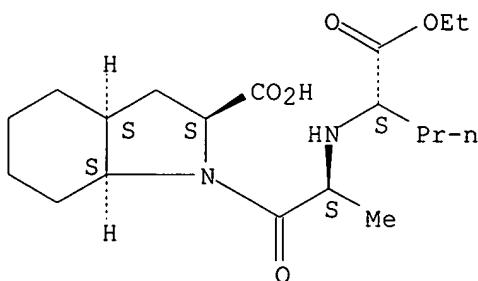
CN 1H-Indole-2-carboxylic acid, 1-[(2S)-2-[[[(1S)-1-  
 (ethoxycarbonyl)butyl]amino]-1-oxopropyl]octahydro-, (2S,3aS,7aS)-, compd.  
 with 2-methyl-2-propanamine (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 82834-16-0

CMF C19 H32 N2 O5

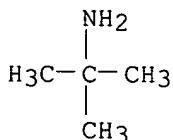
Absolute stereochemistry. Rotation (-).



CM 2

CRN 75-64-9

CMF C4 H11 N



IT 484-42-4

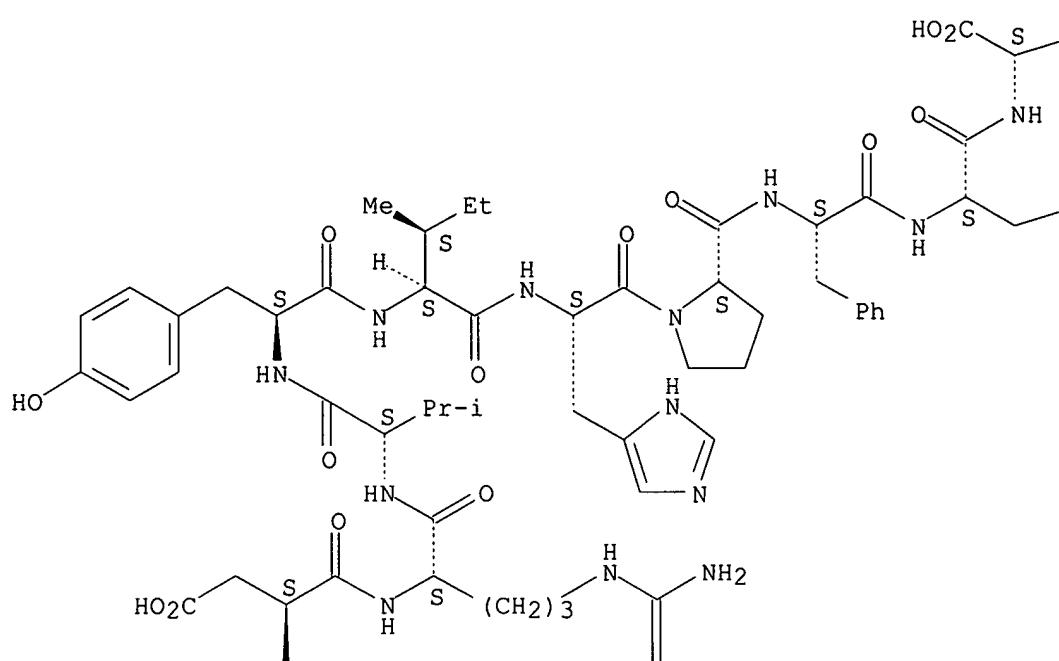
RL: BPR (Biological process); BSU (Biological study, unclassified); BIOL  
 (Biological study); PROC (Process)  
 (process for preparing the novel  $\gamma$  crystalline form of the diuretic  
 perindopril tert-butylamine salt as an intermediate in the preparation of  
 inhibitors of)

RN 484-42-4 HCPLUS

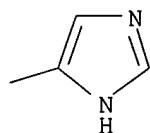
CN Angiotensin I, 5-L-isoleucine- (8CI, 9CI) (CA INDEX NAME)

Absolute stereochemistry.

PAGE 1-A



PAGE 1-B

— Bu-i

PAGE 2-A

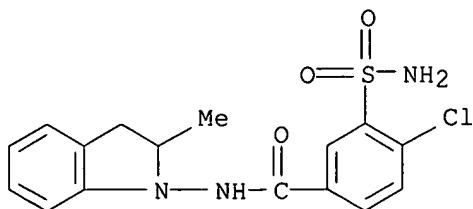


IT 26807-65-8P, Indapamide

RL: PNU (Preparation, unclassified); PREP (Preparation)  
 (process for preparing the novel  $\gamma$  crystalline form of the diuretic  
 perindopril tert-butylamine salt for the preparation of)

RN 26807-65-8 HCAPLUS

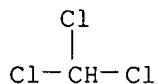
CN Benzamide, 3-(aminosulfonyl)-4-chloro-N-(2,3-dihydro-2-methyl-1H-indol-1-yl)-(9CI) (CA INDEX NAME)



IT 67-66-3, Chloroform, uses 141-78-6, Ethyl acetate, uses  
 RL: NUU (Other use, unclassified); USES (Uses)  
 (solvent; process for preparing the novel  $\gamma$  crystalline form of the  
 diuretic perindopril tert-butylamine salt)

RN 67-66-3 HCPLUS

CN Methane, trichloro- (9CI) (CA INDEX NAME)



RN 141-78-6 HCPLUS

CN Acetic acid ethyl ester (8CI, 9CI) (CA INDEX NAME)

Et-O-Ac